National Institute of Standards & Technology

Certificate

Standard Reference Material 4967A
Radium-226 Radioactivity Standard

This Standard Reference Material (SRM) consists of radioactive radium-226 chloride (and its radioactive decay products), non-radioactive barium chloride and hydrochloric acid dissolved in 5 mL of distilled water. The solution is contained in a flame-sealed NIST borosilicate-glass ampoule. The SRM is intended for the calibration of instruments and for the monitoring of radiochemical procedures.

Radiological Hazard: The SRM ampoule contains radium-226 with an activity of approximately 13 kBq. Radium-226 decays by alpha-particle emission. The progeny of radium-226 have a total activity of approximately 95 kBq and decay by alpha- and beta-particle emission. None of the alpha or beta particles escape from the SRM ampoule. During the decay process, X-rays and gamma rays with energies from 11 kev to 2.5 MeV are also emitted. Most of these photons escape from the SRM ampoule and can represent a radiation hazard. Approximate unshielded dose rates at several distances (as of the reference time) are given in note [a]*. Gaseous radon-222 will escape from the ampoule when it is opened. The SRM should be used only by persons qualified to handle radioactive material.

Chemical Hazard: The SRM ampoule contains hydrochloric acid (HCl) with a concentration of 1.0 mole per liter of water. The solution is corrosive and represents a health hazard if it comes in contact with eyes or skin. If the ampoule is to be opened to transfer the solution, the recommended procedure is given on page 2. The ampoule should be opened only by persons qualified to handle both radioactive material and strong acid solution

Storage and Handling: The SRM should be stored and used at a temperature between 5 °C and 65 °C. The solution in an unopened ampoule should remain stable and homogeneous until at least September 2013. The ampoule (or any subsequent container) should always be clearly marked as containing radioactive material. If the ampoule is transported it should be packed, marked, labeled, and shipped in accordance with the applicable national, international, and carrier regulations. The solution in the ampoule is a dangerous good (hazardous material) because of both the radioactivity and the strong acid.

Preparation: This Standard Reference Material was prepared in the Physics Laboratory, Ionizing Radiation Division, Radioactivity Group, M.P. Unterweger, Acting Group Leader. The overall technical direction and physical measurements leading to certification were provided by R. Collé and P. Volkovitsky of the Radioactivity Group. Statistical consultation was provided by S.D. Leigh of the NIST Statistical Engineering Division. The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program.

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*Notes and references are on pages 5 to 7.

Recommended Procedure for Opening the SRM Ampoule

1) If the SRM solution is to be diluted, it is recommended that the diluting solution have a composition comparable to that of the SRM solution.

2) Wear eye protection, gloves, and protective clothing and work over a tray with absorbent paper in it. Work in a fume hood. In addition to the radioactive material, the solution usually contains strong acid or base and is corrosive.

3) Shake the ampoule to wet all of the inside surface of the ampoule. Return the ampoule to the upright position.

4) Check that all of the liquid has drained out of the neck of the ampoule. If necessary, gently tap the neck to speed the process.

5) Holding the ampoule upright, score the narrowest part of the neck with a scribe or diamond pencil.

6) Lightly wet the scored line. This reduces the crack propagation velocity and makes for a cleaner break.

7) Hold the ampoule upright with a paper towel, a wiper, or a support jig. Position the scored line away from you. Using a paper towel or wiper to avoid contamination, snap off the top of the ampoule by pressing the narrowest part of the neck away from you while pulling the tip of the ampoule towards you.

8) Transfer the solution from the ampoule using a pycnometer or a pipet with dispenser handle. NEVER PIPETTE BY MOUTH.

9) Seal any unused SRM solution in a flame-sealed glass ampoule, if possible, to minimize the evaporation loss.

See also reference [4]*.

*Notes and references are on pages 5 to 7.

Certified values

Radionuclide	**Radium-226**
Reference time	**1200 EST, 01 September 2003**
Massic activity of the solution [b]*	**2482 Bq·g^{-1}**
Relative expanded uncertainty (*k*=2)	**1.20%** [c] [d]
Solution mass	**(5.086 ± 0.003) g** [e]
Solution density	**(1.017 ± 0.002) g·mL^{-1} at 21 °C** [e]

Uncertified values

Physical Properties:			
Source description	Liquid in flame-sealed NIST borosilicate-glass ampoule		
Ampoule specifications	Body outside diameter Wall thickness Barium content Lead-oxide content Other heavy elements	(16.5 ± 0.5) mm (0.60 ± 0.04) mm Less than 2.5% Less than 0.02% Trace quantities	
Chemical Properties:			
Solution composition	Chemical Formula	Concentration (mol·L^{-1})	Mass Fraction (g·g^{-1})
	H_2O HCl $BaCl_2$ $^{226}RaCl_2$	54 1.0 4×10^{-4} 3×10^{-7}	0.96 0.04 8×10^{-5} 9×10^{-8}
Radiological Properties:			
Photon-emitting impurities	None detected [f]		
Half lives used	Radium-226: (1600 ± 7) a [g] [5] Radon-222: (3.8235 ± 0.0003) d [g] [5]		
Calibration method and measuring instrument(s)	Gravimetric dilution of SRM 4963, confirmed by comparison with solution standards, and derivatives thereof, from the NBS/NIST "1947 (1967 recalibrated) series" of radium-226 solution standards. The mass of radium-226 in these solution standards had previously been determined by comparison with the U.S. National Standards for radium-226. Conversion from mass of radium-226 to activity of radium-226 was done using the half life of radium-226 shown above. [h]		

*Notes and references are on pages 5 to 7.

EVALUATION OF THE UNCERTAINTY OF THE MASSIC ACTIVITY [c] [d]*

Input Quantity x_i, the source of uncertainty (and individual uncertainty components where appropriate)	Method Used To Evaluate $u(x_i)$, the standard uncertainty of x_i (A) denotes evaluation by statistical methods (B) denotes evaluation by other methods	Relative Uncertainty Of Input Quantity, $u(x_i)/x_i$, (%) [i]	Relative Sensitivity Factor, $\lvert \partial y/\partial x_i \rvert \cdot (x_i/y)$ [j]	Relative Uncertainty Of Output Quantity, $u_i(y)/y$, (%) [k]
Calibration of the "1947 (1967 recalibrated) series" of radium-226 solution standards in terms of mass of radium-226 [h]	Estimated (B)	0.34	1.0	0.34
Ratio of the mass of radium-226 in SRM 4967A to the mass of radium-226 in the "1947 (1967 recalibrated) series" of radium-226 solution standards	Weighted mean of the ratios obtained using seven different comparisons (B)	0.15	1.0	0.15
Corrections for the decay of radium-226	Standard uncertainty of the radium-226 half-life (A) [m]	0.44	0.016 [n]	0.007
Gravimetric measurements	Estimated (B)	0.10	1.0	0.10
Conversion of radium-226 mass to activity [p]	Standard uncertainty of the radium-226 half-life (A) [q]	0.44	1.0	0.44
Photon-emitting impurities	Limit of detection (B) [r]	100.	0.0001	0.01
Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$, (%)				0.6
Coverage Factor, k				x 2
Relative Expanded Uncertainty of the Output Quantity, U/y, (%)				1.2

*Notes and references are on pages 5 to 7.

NOTES

[a] The Sievert is the SI unit for dose equivalent. See reference [1]. One μSv is equal to 0.1 mrem.

Distance from Ampoule (cm):	1	10	100
Approximate Dose Rate (μSv/h):	5	0.4	-

[b] **Massic activity** is the preferred name for the quantity activity divided by the total mass of the sample. See reference [1].

[c] The reported value, y, of massic activity (activity per unit mass) at the reference time was not measured directly but was derived from measurements and calculations of other quantities. This can be expressed as $y = f(x_1, x_2, x_3, \ldots x_n)$, where f is a mathematical function derived from the assumed model of the measurement process. The value, x_i, used for each input quantity i has a **standard uncertainty**, $u(x_i)$, that generates a corresponding uncertainty in y, $u_i(y) \equiv |\partial y/\partial x_i| \cdot u(x_i)$, called a **component of combined standard uncertainty** of y. The **combined standard uncertainty** of y, $u_c(y)$, is the positive square root of the sum of the squares of the components of combined standard uncertainty. The combined standard uncertainty is multiplied by a **coverage factor** of $k = 2$ to obtain U, the **expanded uncertainty** of y.

Since it can be assumed that the possible estimated values of the massic activity are approximately normally distributed with approximate standard deviation $u_c(y)$, the unknown value of the massic activity is believed to lie in the interval $y \pm U$ with a level of confidence of approximately 95 percent.

For further information on the expression of uncertainties, see references [2] and [3].

[d] The value of each component of combined standard uncertainty, and hence the value of the expanded uncertainty itself, is a best estimate based upon all available information, but is only approximately known. That is to say, the "uncertainty of the uncertainty" is large and not well known. This is true for uncertainties evaluated by statistical methods (e.g., the relative standard deviation of the standard deviation of the mean for the massic response is approximately 50%) and for uncertainties evaluated by other methods (which could easily be over estimated or under estimated by substantial amounts). The unknown value of the expanded uncertainty is believed to lie in the interval $U/2$ to $2U$ (i.e., within a factor of 2 of the estimated value).

[e] The stated uncertainty is two times the standard uncertainty.

[f] Estimated limits of detection for photon-emitting impurities, as of the reference time, expressed as massic photon emission rates, are:
6×10^{0} $s^{-1} \cdot g^{-1}$ for energies between 22 and 182 keV,
3×10^{0} $s^{-1} \cdot g^{-1}$ for energies between 190 and 347 keV,
8×10^{-1} $s^{-1} \cdot g^{-1}$ for energies between 356 and 1455 keV, and
3×10^{-1} $s^{-1} \cdot g^{-1}$ for energies between 1465 and 2750 keV,
provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of radium-226 and progeny.

[g] The stated uncertainty is the standard uncertainty.

[h] For further details on NBS/NIST radium series calibrations refer to reference [6]. The 1967 recalibrations of the "1947 series" and of the "1957 series" were made using pressurized "4π"γ ionization chamber (PIC) "A".

The master solution for SRM 4967A was directly compared with the "1947 (1967 recalibrated) series" of radium-226 solution standards using PIC "A", and was compared with solutions of the "1992 series" of radium solution standards (SRM 4967) using PIC "A", pulse-ionization-chamber radon analyses (see references [7] and [8]), and germanium photon spectrometry.

The radium-226 in SRM 4967A was chemically purified approximately 55 years ago. The lead-210 and its daughter radionuclides are not in equilibrium.

[i] Relative standard uncertainty of the input quantity x_i.

[j] The relative change in the output quantity y divided by the relative change in the input quantity x_i. If $|\partial y/\partial x_i|\cdot(x_i/y) = 1.0$, then a 1% change in x_i results in a 1% change in y. If $|\partial y/\partial x_i|\cdot(x_i/y) = 0.05$, then a 1% change in x_i results in a 0.05% change in y.

[k] Relative component of combined standard uncertainty of output quantity y, rounded to two significant figures or less. The relative component of combined standard uncertainty of y is given by $u_i(y)/y \equiv |\partial y/\partial x_i|\cdot u(x_i)/y = |\partial y/\partial x_i|\cdot(x_i/y)\cdot u(x_i)/x_i$. The numerical values of $u(x_i)/x_i$, $|\partial y/\partial x_i|\cdot(x_i/y)$, and $u_i(y)/y$, all dimensionless quantities, are listed in columns 3, 4, and 5, respectively. Thus, the value in column 5 is equal to the value in column 4 multiplied by the value in column 3. The input quantities are independent, or very nearly so. Hence the covariances are zero or negligible.

[m] The relative standard uncertainty of $\lambda\cdot t$ is determined by the relative standard uncertainty of λ (i.e., of the half life). The relative standard uncertainty of t is negligible.

[n] $|\partial y/\partial x_i|\cdot(x_i/y) = |\lambda\cdot t|$

[p] The U.S. National Standards for radium-226 are certified in terms of mass of radium-226, as were all radium-226 SRMs prior to the "1992 series". Beginning with the "1992 series", radium-226 solution SRMs are now certified in terms of the massic activity of radium-226.

[q] The relative standard uncertainty of the activity of radium-226 per unit mass of radium-226 is determined by the relative standard uncertainty of λ (i.e., of the half life). The relative standard uncertainties of the atomic weight of radium-226 and of Avogadro's number are negligible.

[r] The standard uncertainty for each undetected impurity that might reasonably be expected to be present is estimated to be equal to the estimated limit of detection for that impurity, i.e. $u(x_i)/x_i$ = 100%. $|\partial y/\partial x_i|\cdot(x_i/y)$ = {(response per Bq of impurity)/(response per Bq of Ra-226)}·{(Bq of impurity)/(Bq of Ra-226)}. Thus $u_i(y)/y$ is the relative change in y if the impurity were present with a massic activity equal to the estimated limit of detection.

REFERENCES

[1] International Organization for Standardization (ISO), *ISO Standards Handbook - Quantities and Units*, 1993. Available from Global Engineering Documents, 12 Inverness Way East, Englewood, CO 80112, U.S.A. Telephone 1-800-854-7179.

[2] International Organization for Standardization (ISO), *Guide to the Expression of Uncertainty in Measurement*, 1993 (corrected and reprinted, 1995). Available from Global Engineering Documents, 12 Inverness Way East, Englewood, CO 80112, U.S.A. Telephone 1-800-854-7179.

[3] B. N. Taylor and C. E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, 1994. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20407, U.S.A.

[4] National Council on Radiation Protection and Measurements Report No. 58, *A Handbook of Radioactivity Measurements Procedures*, Second Edition, 1985. Available from the National Council on Radiation Protection and Measurements, 7910 Woodmont Avenue, Bethesda, MD 20814 U.S.A.

[5] Evaluated Nuclear Structure Data File (ENSDF), September 2003.

[6] W.B. Mann, L.L. Stockman, W.J. Youden, A. Schwebel, P.A. Mullen and S.B. Garfinkel, Preparation of New Solution Standards of Radium, *Journal of Research of the National Bureau of Standards* **62** (1959) 21-26.

[7] R. Collé, J.M.R. Hutchinson and M.P. Unterweger, The NIST Primary Radon-222 Measurement System, *Journal of Research of the National Institute of Standards and Technology* **95** (1990) 155-165.

[8] J.M.R. Hutchinson, J. Cessna, R. Collé and P. Hodge, An International Radon-In-Air Measurement Intercomparison Using a New Transfer Standard, *Applied Radiation and Isotopes* **43** (1992) 175-189.